Manufacturability of Gelcast Ceramics for Advanced Heat/Gas Turbine Engines

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Abstract

Steps in gelcasting that enhance the manufacturability of gelcast ceramics have been examined. Gelcast alumina plates of varying thickness were dried under conditions that reduced the drying times to under 40 hours, about half the usual drying time. Although parts up to 2 inches thick were dried, only 0.5 inch plates survived sintering and their densities varied from 97.7 to 99.7 % of theoretical density. Nondestructive evaluation showed flaws 100 - 200 μm in diameter uniformly distributed throughout the plates which had Young's and shear moduli comparable to literature values. Green machining of dried gelcast parts, which is applicable to rapid prototyping or flexible manufacturing, has been investigated. By introducing additives to the slips for gelcasting, green-machined 1 inch thick gelcast blocks that were successfully sintered to full density. The mold surface interaction with gelcasting slip was investigated. Molds which are unsuitable for gelcasting inhibit the gelation of the monomers at the interface.

Introduction

Gelcasting, a generic forming process developed at the Oak Ridge National Laboratory has been described,[1,2] although its development continues.[3] Its applicability not only to ceramics[1,2] but also to powder metallurgy has been demonstrated.[4] For forming large, complex-shaped ceramic parts, gelcasting has proven advantages over comparable processes such as slip casting and injection molding.[5] At this forum last year, the special advantages gelcasting has over slip casting for the manufacture of silicon nitride turbomachinery components was reported.[6] However, the development of the process for the manufacture of ceramic components for diesel engines or gas turbine engines, requires the adaption and scale-up of gelcasting to high volume production. This presentation deals with some of the studies relevant to the manufacturability of gelcast ceramic components.

Drying Studies

High-volume production requires increasing the speed of each of the steps in the forming process. The slowest step in gelcasting is the drying process. Consequently, the drying of wet gelcast parts has been studied in order to reduce the time it takes.

Past experience has shown that, to avoid warpage or cracking, the initial drying should be carried out at high relative humidity until the part stops shrinking when there is particle-to-particle contact. The rate of drying can then be speeded up by either lowering the humidity at constant temperature or raising the temperature to reduce the humidity.

A series of drying experiments was performed with 4 inches x 4 inches Al_2O_3 plates 0.5 inch, 1 inch, or 2 inches thick, respectively, under the three drying schedules shown in Table 1. As the schedules indicate, the drying was started at high relative humidity (92 %) and low temperature (25°C) and then the conditions were changed to increase the rate of drying. The plates were dried, then the binders were burned out, and if they survived burnout, were sintered. Table 2 shows the results of this study. After drying, all the 0.5 inch plates survived binder burnout whereas all the 1 inch plates cracked into several pieces. A 2 inch plate not shown in the table also cracked into several pieces during the burnout. This indicates that plates 1 inch or thicker cannot be dried successfully using any of the three drying schedules. Other experiments at different drying schedules have confirmed this conclusion. However, this does not imply that thick gelcast parts cannot be dried; AlliedSignal Ceramic Components routinely dries parts thicker than 2 inches. In this study, the appropriate drying schedules for thick parts have not been identified.

Table 1: Three Schedules for Drying Gelcast Parts

Drying Schedule I

Time,h	RH,%	Temp,°C
0	92	25
0.5	92	25
10.5	92	25
11.5	75	25
21.5	75	25
22.5	50	25
33	50	25

Drying Schedule II

Time, h	RH, %	Temp,°C
0	92	25
0.5	92	35
1.5	92	35
11.5	92	35
12.5	75	35
22.5	75	35
23.5	50	35
33.5	50	35
34.5	50	25
35.5	50	25

Drying Schedule III

Time, h	RH, %	Temp,°C
0	92	25
0.5	92	25
1.5	92	35
11.5	92	35
12.5	75	35
22.5	75	35
23.5	50	35
24.5	50	50
34.5	50	50
35.5	50	25
36.5	50	25

As Table 2 shows only one of the 0.5 inch plates did not survive sintering, probably because water condensate dropped on it as it dried. It cracked into two pieces only. The sintered parts attained high density, 97.7 to 99.7% of theoretical density. After sintering, a large flaw that looked like a water drop, which was not visible on the dried plate, appeared on plate #1.

Table 2: Detailed Data on Dried Alumina Plates

Run #	Drying Sched.	Size of plate	Premix	% of H20 Lost	Survived Burnout	Survived Firing	% T. Density
1	I	.5" x 4" x 4"	MAM/MBAM	98	Yes	Yes	97.7
3	II	.5" x 4" x 4"	MAM/MBAM	97.3	Yes	Yes	97.8
5	I	.5" x 4" x 4"	MAM/PEG	94.1	Yes	Yes	97.7
6	II	.5" x 4" x 4"	MAM/PEG	82.5-cond.	Yes	No	99.7
7	II	.5" x 4" x 4"	MAM/MBAM	99	Yes	Yes	98.4
9	III	.5" x 4" x 4"	MAM/PEG	91.4	Yes	Yes	99.7
11	III	.5" x 4" x 4"	MAM/MBAM	98.8	Yes	Yes	99.6
2	I	1" x 4" x 4"	MAM/MBAM	101.4	No	-	-
8	II	1" x 4" x 4"	MAM/MBAM	98.2	No	-	-
10	I	1" x 4" x 4"	MAM/PEG	71.3	No	-	-
12	III	1" x 4" x 4"	MAM/MBAM	84.9	No	-	-
13	II	1" x 4" x 4"	MAM/PEG	74.8-crack	No	-	-

Defect-free Gelcasting

Ideally, ceramic components for aerospace and automotive needs should be completely defect-free. In practice, this is impossible; consequently, a critical objective in the development of a process for high volume manufacturing, is to minimize the defects in the ceramic part. To this end, three 0.5 inch thick gelcast plates (#1, #9, and #11 in Table 2) were polished and subjected to nondestructive evaluation (NDE). The NDE was done by ultrasonic testing in three modes: flaw test, back-surface test, and surface-wave test.

The flaw test in which the ultrasonic waves penetrated the samples indicated that the range of flaws sizes in samples # 9 and #11 was 100 - 200 μm. The flaw test also showed clearly the large flaw from the water drop that condensed on sample #1. In addition, the flaw test indicated that the flaws were distributed fairly uniformly over the samples even for the sample #1 with its large water drop flaw. The surface-wave tests showed the polish lines and the water drop mark on the surfaces of the plates. From the NDE, the Young's and shear moduli, and the Poisson's ratio were calculated as shown in Table 3. These values are similar to those reported in the literature for alumina. These results demonstrated that NDE is useful for both quantitative and qualitative evaluation of gelcast parts which were found to produce defects comparable to those from other ceramic forming processes.

Table 3: NDE Data on Alumina Plates

Plate #	(E) Young's Modulus, GPa	(G) Shear Modulus, GPa	(v) Poisson's Ratio
1	380	154	0.232
9	403	163	0.236
11	401	162	0.238

Green Machining - Rapid Prototyping/Flexible Manufacturing

A major objective of the Advanced Automotive Materials Program is to reduce the cost of the fabrication of advanced ceramics. Gelcasting produces dried parts with high green body strength which can be machined. Thus, although gelcasting was developed primarily as a near-net-shape forming technique, gelcast parts can be green-machined into more complex shapes using inexpensive tools. This concept has resulted in the investigation of gelcasting for rapid prototyping and flexible manufacturing. Simple blocks of ceramic material are gelcast and dried. A block is green-machined with a computer-controlled three-axis machine into any desired shape. Green machining of gelcast blocks is applicable not only to advanced ceramics but also to bioceramics and powder metallurgy. As a result, the investigation of the rapid prototyping of bioceramics for implants, funded by other sponsors, is being pursued.

A major effort has been directed towards understanding the parameters that affect green machining such as spindle speed, feed and plunge rates, depth of cut, and tool choice for the different ceramics and monomer combinations. Several blocks of gelcast silicon nitride have been successfully green-machined as shown in Fig. 1. However, a critical step in the production of the blocks for green machining is the drying. As was reported earlier, dried gelcast parts 1" or thicker have not survived binder burnout, and this will limit the rapid prototyping of thick parts. To over come this problem, several additives have been introduced into gelcasting slips for the preparation of parts to be green-machined. By using these additives, 1 inch gelcast blocks have been green-machined and successfully sintered. Figure 2 shows gelcast Si_3N_4 parts machined in the green state from a 1 inch thick block which have been fired to full density. Without these additives such parts have not sintered without cracking. The investigation continues to identify the best additives and to optimize the amounts to be included during the slip preparation.

Mold Release - Mold Surface/Gel Interaction

Another important component in the fabrication of advanced ceramics is the mold for complex shapes such as turbine rotors. Currently the molds used are based on the designs for other forming processes such as injection molding or metal casting. The need to design molds based on gelcasting cannot be overemphasized. Fortunately, gelcasting has been carried out successfully in several mold materials such as metal, glass, wax and plastics. The mold release material facilitates the separation of the formed part from the mold. This is important for a high yield manufacturing process, especially where there are thin-thick parts as in turbine rotors. Several mold releases have been tested for the metal molds, usually aluminum, used in gelcasting. A few have proved adequate. Nevertheless, the search for improved mold releases continues.

When the mold is fabricated from certain plastic materials there is incomplete gelation at the mold surface. Thus, the interaction at the mold interface during gelcasting is important, especially for the rapid prototyping of the molds which usually use plastics. Gelation has been studied in two types of mold: (1) glass, and (2) RTV silicone. Glass is good mold material for ceramic gelcasting since the polyacrylamide gels formed in glass molds are fully expanded and completely fill the interior of the mold. RTV silicone, on the other hand, is a poor mold material because the gels cast against RTV are significantly smaller in diameter than the mold and the cast part has a thin fluid of ceramic on its surface.

For the initial studies, the weight of material rinsed from the gel surface in the different molds, has been used as a measure of the extent of polymerization. The weight of material rinsed from the gel surface has been divided by the initial combined weight of the monomers in the polymerizing solution and then converted to percent. The amount of material soluble in the rinses ranged from 2 to 10% of the initial weight of the monomers. Three different RTV molds were used to hold the polymerizing solution. Neither the type of RTV nor the amount of the agent used to cure the mold had any effect on the amount of water-soluble residue on the surface of the gel. However, as shown in Table 4, the amount of dried residue obtained from the glass molds was always less than that from the RTV mold. The studies of the interaction of mold materials and the polymerization process continues. A High Performance Liquid Chromatography (HPLC) will be used to measure the unreacted monomer left in the liquid phase after the gelation reaction is quenched.

Table 4: Comparison of material obtained by rinsing surface gels in various molds.

Residue after drying to constant weight

RTV-60 Mold 0.16% DBT	RTV-60 Mold 0.52% DBT	RTV-30	Glass Beaker # 1	Glass Beaker # 2
6.50%	no data	3.80%	2.20%	2.20%
3.50%	3.10%	no data	2.20%	2.10%
3.60%	3.90%	no data	no data	2.20%

Technology Transfer of Gelcasting

The gelcasting group at ORNL has continued to work closely with AlliedSignal Ceramic Components (CC) which has licensed the process and is pursuing its commercialization for the fabrication of turbomachinery components. CC's presentation at this meeting will demonstrate this association as gelcasting is a major forming technique employed by them in many of the programs. Furthermore, a member of the ORNL group is currently on a two year off-site assignment at CC in Torrance, CA. Another licensee of the process, LoTEC Inc., of Salt Lake City, UT is using gelcasting to fabricate complex-shaped low thermal conductivity ceramic parts.

LoTEC, Inc. is represented at this meeting.

The gelcasting process has also been licensed by Ceramic Magnetics, Inc of Fairfield, NJ, for the manufacture of soft ferrite magnets used in high energy physic. The high green strength of gelcast parts enables them to fabricate large magnetic parts.

Conclusions

The drying time for gelcast plates was reduced to under 40 hours, about half the usual length, by varying the drying temperature and humidity. Although samples up to 2 inches thick were dried successfully, only 0.5 inch plates survived through sintering. These plates were successfully subjected to nondestructive evaluation and had properties similar to those in the literature. Blocks of dried gelcast alumina and silicon nitride have been successfully machined in the green state. By introducing additives into the slip for producing the ceramic blocks for green machining, parts over 0.5" thick have survived sintering. RTV silicone, a poor mold material, produces more unreacted monomers at the mold interface compared to good mold materials such as glass.

The gelcasting technology has been licensed by three companies, two in structural ceramics and one in magnetic ceramics. Because it is a generic forming process, it has been applied to investigations in bioceramics which is funded by other sponsors.

Future Direction

The investigation of the drying of gelcast parts, the slowest step in the process, has implications not only for fabricating ceramic parts directly but also for the green machining of gelcast parts. The study of drying will be carried on for both applications. The production of gelcast parts with acceptable defects and the adaption of gelcasting to rapid prototyping by green machining will continue. However, the most important area that needs attention is the development of molds designed specifically for gelcasting. This is important for the use of gelcasting in high production manufacturing and requires collaboration between engine designers, mold designers, component manufacturers using gelcasting and researchers in gelcasting.

References

1. O. O. Omatete, M. A. Janney & R. A. Strehlow, "Gelcasting - A New Ceramic Forming Process," Am. Ceramic Bulletin, 70 [10]. Oct 1991.
2. A. C. Young, O. O. Omatete, M. A. Janney, & P. A. Menchhofer, "Gelcasting of Alumina," J. of Am. Ceram. Soc., 74 [3] 612-18 (1991).
3. M. A. Janney, O. O. Omatete, C. A. Walls, S. D. Nunn, R. J. Ogle, & G.C. Westmoreland, "Development of Low-Toxicity Gelcasting System," to be submitted to the J. Am. Ceram. Society.
4. M. A. Janney, W. Ren, and S. Viswanathan, "Gelcasting H13 Tool Steel," to be published in **Advances in Powder Metallurgy and Particulate Materials, 1996**, Metals Powder Industries Federation, Princeton, NJ, 1996.
5. O. O. Omatete, M. A. Janney, & S. D. Nunn, "Gelcasting: from laboratory development toward industrial production," accepted for publication in J.